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Ion guiding in alumina capillaries: MCP images of the transmitted ions

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ABSTRACT

Transmission of a few keV impact energy Ne^{6+} ions through capillaries in anodic alumina membranes has been studied with different ion counting methods using an energy dispersive electrostatic spectrometer, a multichannel plate (MCP) array and sensitive current-measurement. In the present work, we focus our attention to the measurements with the MCP array. The alumina capillaries were prepared by electrochemical oxidation of aluminium foils. For the present experiments guiding of 3–6 keV Ne^{6+} ions has been studied in two samples with capillary diameter of about 140 nm and 260 nm and with capillary length of about 15 μm. At these energies, the ions have been efficiently guided by the capillaries up to few degrees tilt angle. In this work, we compare the results obtained by the energy dispersive spectrometer to those studied by the MCP array.

1. Introduction

Insulating nanocapillaries have attracted considerable attention since the discovery of capillary guiding [1–8]. Ions with a few keV kinetic energy are efficiently transmitted through the capillaries of thin insulating foils mostly in their initial charge state, and the transmitted ions are directed along the capillaries with a narrow angular distribution. There is significant transmission even if the capillaries are tilted by large angles, i.e. when there is no geometrical transparency for straight line trajectories. Due to these properties, insulating nanocapillary arrays might find numerous applications, e.g. in guiding, directing and focusing slow ion beams in nanoscale devices. They might be used for irradiating single cells and writing on charge sensitive surfaces. Collecting experimental data with capillary samples made of different kinds of materials is of a great importance for the understanding of this self-organizing phenomenon [1–9].

In the present work we study the transmission of Ne^{6+} ions through Al_2O_3 capillaries. The capillaries were prepared at UCL using the self-ordering phenomena during a two-step anodization process. The details of the preparation are given in [9]. We measured angular and charge state distributions of the transmitted ions with two techniques using a position sensitive MCP detector and an electrostatic spectrometer. The measured angular distributions agree well for the different techniques. With the MCP detector, however, significant ultra violet (UV) photon emission has been observed stemming partly from the capillaries. For this reason special care has been taken to separate charged and neutral particles.

2. Experiment

The experiments were carried out at the Institute of Nuclear Research (ATOMKI), Debrecen. Ions were provided by a 14.5 GHz electron cyclotron resonance (ECR) ion source [10]. The typical beam current for 3 keV Ne^{6+} was about 300 pA. For 6 keV Ne^{6+}, it was as large as 500 pA. A collimator set of two 1 mm diaphragms at a distance of 205 mm restricted the beam divergence to ±0.3° from axis at the end of the beamline.

The schematic view of our experimental apparatus can be seen in Fig. 1. The sample holder with the samples was mounted in the middle of a vacuum chamber with three axial and one rotational freedom. Separately, the spectrometer was mounted on a rotating table to measure the transmitted intensities at different observation angles. The rotational axes of the target samples and the rotating table for the spectrometer were carefully adjusted to match and cross the beam axis with an accuracy of about 0.1 mm. The incidence angle of the ions was changed by tilting the target samples. Two-dimensional angular distributions were recorded by the MCP detector placed behind the target samples. On the sensitive surface of the detector a voltage of −100 V was applied in order to get rid of counts due to low energy electrons. The MCP detector was shielded by a metallic cover, while in the front of it a grounded mesh was ap-

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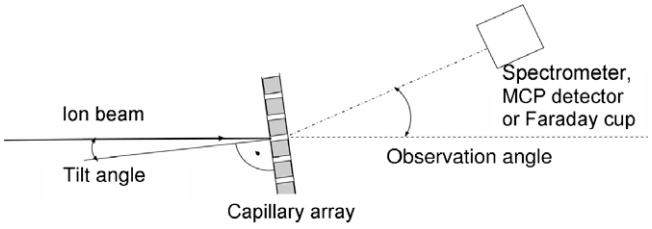

Fig. 1. Schematic view of the experimental set-up.

plied. During the exposition of the MCP images, the current incident on the target was reduced to few pA in order to avoid overloading the detector. When the electrostatic spectrometer was used the ions transmitted through the capillary samples were detected by a channeltron after passing through a 45° parallel-plate electrostatic analyzer. The diameter of its opening aperture was 1.5 mm, which corresponds to an acceptance angle of 0.7°. The ions are separated by the spectrometer according to their energy-to-charge ratio with a resolution of 7%, i.e. the different charge states of Ne are well separated in the experiments. This arrangement allowed us to measure the angular distributions in the plain defined by the incidence angle. The angular distribution of the primary beam was measured by the spectrometer and the full width at half maximum (FWHM) was found to be 0.82°. This is in accordance with the acceptance angle and the maximum beam divergence.

The target samples were nearly circularly shaped membranes of nanochanneled Al_2O_3 with a diameter of about 8 mm. The membranes have been prepared suspended on aluminium frames for an easy handling during experimental investigations. The thickness of the samples was about 15 μm and the capillaries were ordered in a honeycomb structure (see SEM images in Fig. 2). Two different samples were used in the present experiments. The average inner capillary diameters of the samples are about 140 and 260 nm, respectively. In order to prevent macroscopic charging up of the samples, a niobium (Nb) layer of about 20 nm thickness was deposited on both sides of the membranes by dc-sputtering. The SEM image for the 260 nm sample has been made before dc-sputtering, while the 140 nm sample is shown after dc-sputtering, i.e. covered by the conductive Nb layer.

Orienting the capillaries parallel to the ion beam was a complicated process in general, due to the macroscopic ripples on the membranes. At different positions of the samples the capillary directions slightly differed due to those ripples in the order of ±1°. We searched for an optimum position, and aligned the samples before each set of measurements by seeking the minimal deviation and maximal transmission of the transmitted ions in order to find the correct zero point of our tilt angle scale.

Due to unavoidable irregularities in the nanochannel growing process of anodized Al_2O_3 capillary samples, deviations from the ideal, parallel-arranged structure of the individual capillary axes are present in the used samples. Non-parallelism may strongly influence the guiding parameters, especially the width of the angular distribution [11,12]. In order to study the geometric properties of the samples, we measured the transmission of 90 keV Ne^{6+} ions by a current-measurement technique. At this large energy no significant guiding has been found. The transmission was mainly determined by the geometry. Ions have been transmitted only in the zero degree direction, but in a rather wide range of the tilt angle. The fact that we observed significant transmission for tilted samples up to a few degrees indicates relatively broad angular distributions for the capillary directions. The estimated widths of the angular distributions from the experiments are about 1.1° and 2.4° at half maximum for the 140 and 260 nm capillary samples, respectively. These values are typical for capillary samples prepared by anodization.

3. Results and discussion

In Fig. 3, the angular distributions of the transmitted ions, measured by the electrostatic spectrometer, are shown for the 260 nm sample. The observed angular distributions are fitted by Gaussian functions. The centroids of the curves shift according to the tilt angles of the sample. This is a clear sign for the ion guiding. The overall transmitted intensities can be estimated from the one dimensional angular distributions assuming azimuthal symmetry. This assumption is verified by the two-dimensional MCP images (see below). At ~4.4° tilt angle, the overall transmission drops to

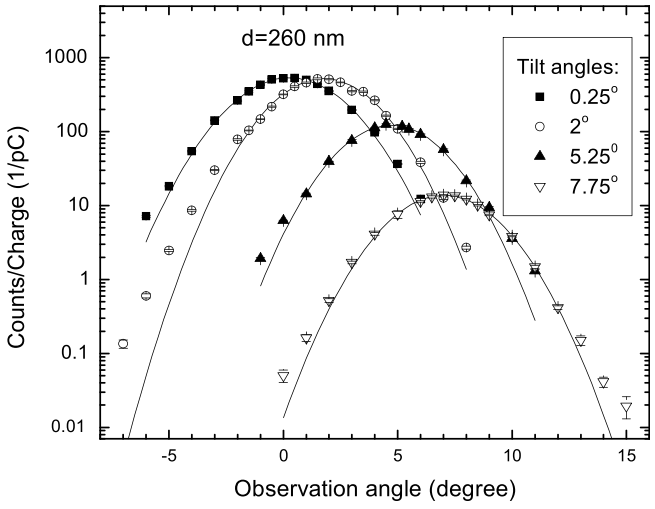

Fig. 3. Angular distributions of 3 keV Ne^{6+} ions transmitted through nanocapillaries of Al_2O_3 with diameters of $d = \sim 260$ nm measured by the spectrometer. The capillaries were tilted as indicated in the figure. Solid lines represent Gaussian fits to the data. The signal was normalized to integrated charge measured on the target sample (not corrected for secondary electron emission).

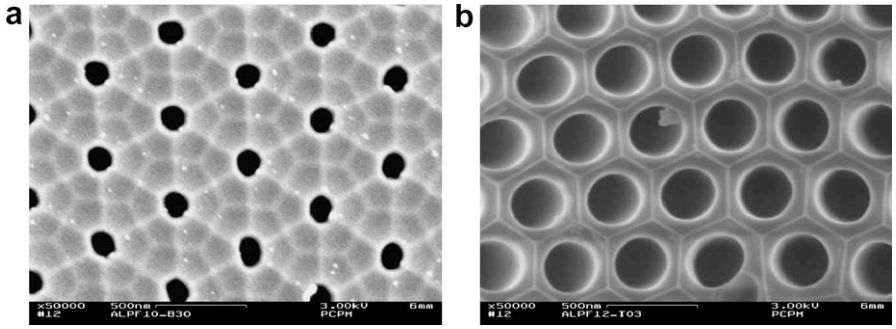

Fig. 2. SEM images of the Al_2O_3 nanocapillary array samples. The diameters of the capillaries are (a) $d = \sim 140$ nm, (b) $d = \sim 260$ nm.

1/*e* fraction of the maximum value. Discounting the effect of the angular distribution of the capillary axes of 2.4° width in order to obtaining the value for ideally parallel capillaries, this angle is reduced to 3.7°. For PET capillaries significantly better [1–4], but for Al_2O_3 [7] and SiO_2 [6] capillaries smaller guiding abilities were reported earlier. This indicates that the guiding ability is strongly dependent on the material properties. Since there were differences in sample geometry and projectile beam properties in the different experiments, direct comparison of the results, however, is not possible. A spectrum of the transmitted ions according to the energy-to-charge ratio is shown in Fig. 4. In accordance with previous observations with insulating capillaries [1–4], the majority of the outgoing ions are in their initial charge state (6+ in our case). The total fraction of transmitted ions with other charge state is less than 3%. This indicates that the great majority of the transmitted ions do not suffer close collisions with the capillary walls.

The use of a MCP detector has significant advantages. For example, two-dimensional angular distributions of the transmitted ions can be recorded in one step. However, in the case of a MCP detector, different charge states can be separated only by applying a transversal electric field in front of the detector. At the early stage of the experiments, significant amount of non-deflectable particles were detected by the MCP detector in the primary beam. Such particles can be photons or projectile ions neutralized by charge exchange processes. Since the charge exchange with the background gases is negligible at our vacuum level ($\sim 10^{-7}$ mbar), the neutral particles are most likely UV photons. They originate from the beamline, created by ions of different charge states impacting at the walls at the analyzing magnet. Charge state selected, analyzed ions may also hit collimator slits or other surfaces in the beamline. In order to separate incoming photons and ions, an electrostatic deflector system was applied after the collimator (see Fig. 5).

In Fig. 6(a), a MCP image is shown. Here the sample foil filled only half of a circular opening on the sample holder (see the right side of Fig. 5). The UV photons passed the sample holder above the sample foil, and hit the MCP detector directly. The ion beam, which was shifted by 4 mm downwards by the deflector system, hit the untilted target sample of 140 nm diameter capillaries. The lowest broad spot is due to ions transmitted trough the sample. It has a width of about 4° at half maximum. Similar angular distributions were measured with the electrostatic spectrometer (see Fig. 3). The upper, sharp and intense spot is due to the photons, which directly hit the detector. There are some less intense spots even above it. Those are the ions, which are scattered at the deflector plates and pass through the diaphragms. Such ions reach the detector freely. Though their fraction seems to be significant, one has to note, that their contribution is very low, it is in the order of 0.1% of the incoming ion beam.

In Fig. 6(b) the same scattering pattern is shown, but with a horizontal electric field applied in front of the detector in order to separate different charge states. As a result, the spots due to ions are deflected toward right. The magnitude of the deflection depends on their charge state. The small, bright spot due to the photons remains in the same position. The most important fact is that only one charge state is visible for the ions transmitted through the foil. This is in complete accordance with the observations made by the

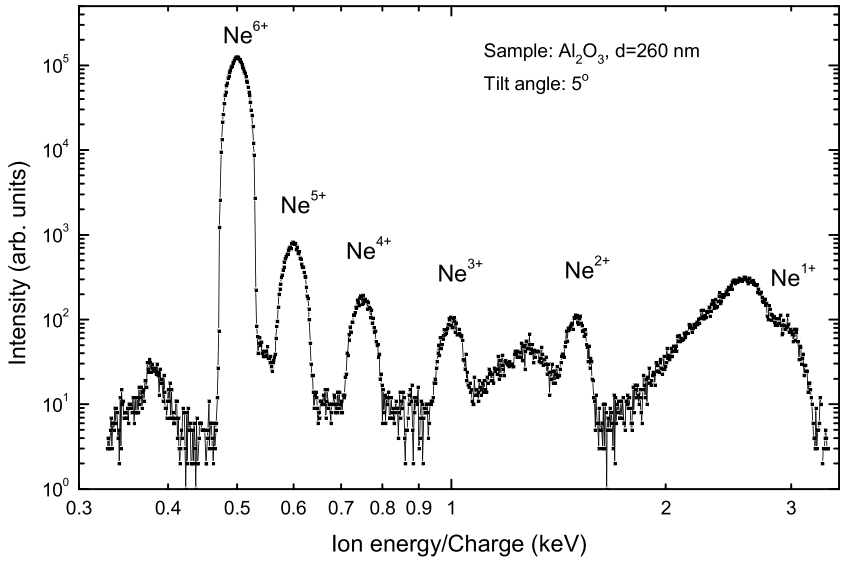

Fig. 4. Charge state distribution of the ions transmitted through the 260 nm sample tilted at 5° measured by the electrostatic spectrometer. The incident beam was of 3 keV Ne^{6+} ions. Non labeled peaks (<1%) are most likely due to beam impurities.

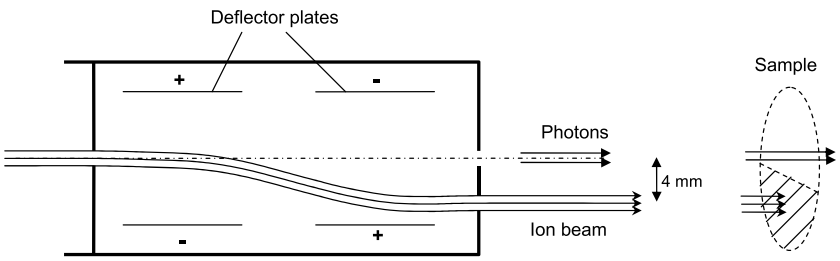

Fig. 5. Schematic view of the ion beam deflector for separating the incoming ion and photon beams, and the half-foil sample, which was used for testing it

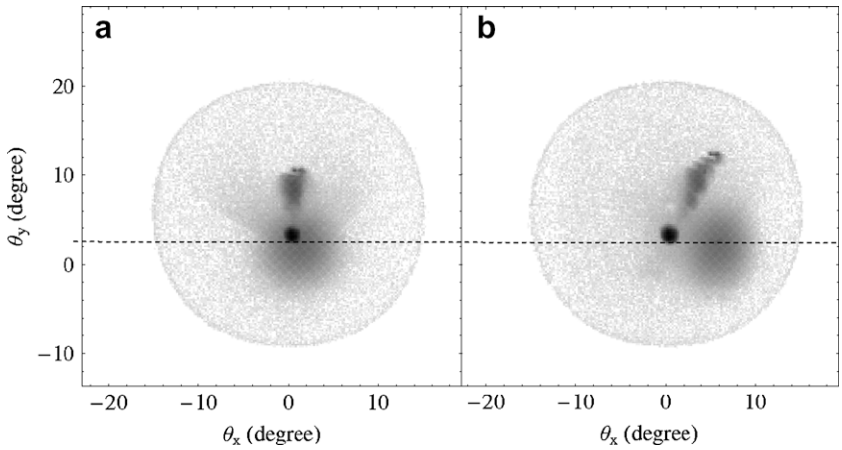

Fig. 6. MCP images of the two-dimensional angular distribution of 3 keV Ne^{6+} ions transmitted through the 140 nm sample foil (for the arrangement, see Fig. 5) at 0° tilt angle. *Panel (a)*: without a deflection field, *Panel (b)*: with a horizontal electrostatic deflection field applied in front of the detector in order to separate different charge states. The upper edge of the capillary foil (see the sample in Fig. 5) is indicated with the dashed line.

spectrometer, that the overwhelming majority of the ions transmitted trough the capillaries are in their initial charge state (see Fig. 4).

In Fig. 7, MCP images are shown for 6 keV Ne^{6+} ions transmitted through the 260 nm capillary samples at different tilt angles. The photon beam was blocked in this case. There is a clear indication of the capillary guiding since the maxima of the transmissions nearly coincide with the tilt angles (read at the observation angle axes of the upper panels). Note that the MCP was always rotated to be parallel with the tilted sample foil, i.e. into the position where the ion distribution was expected to be located in the center of the detector. The nice circular shapes of the spots indicate that the angular distributions are azimuthally symmetric. In the lower panels, images with charge state separation are presented (positive ions are always deflected to the right direction). At 0° tilt angle only one charge state is visible as in the previous case for the 140 nm sample. For tilted samples, significant emission of non-deflected particles appears beside the Ne^{6+} ions. Again, this emission is most likely due to photons, because there is no emission of ions with lower charge states are observed, as it is clearly seen in Fig. 7. We would like to emphasis here that it is unlikely to observe only 6-fold

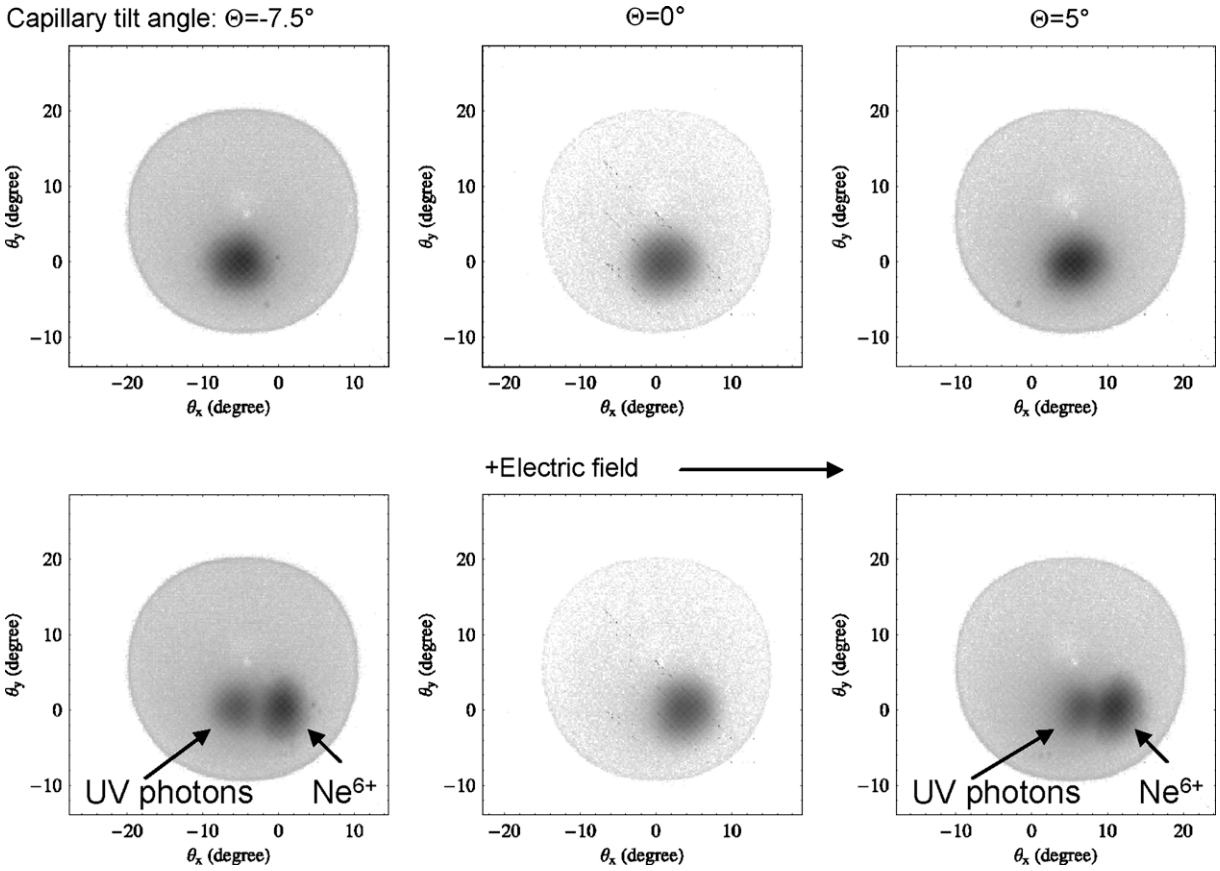

Fig. 7. MCP images of two-dimensional angular distributions of 6 keV Ne^{6+} ions transmitted trough the 260 nm sample at different tilt angles (upper panels). A horizontal electric field was applied in front of the MCP detector in order to separate different charge states (lower panels).

charged ions and fully neutralized ions, without any trace of partial neutralization processes. It is supported by the recent observation of neutralized ions originating from polymer capillaries, where they appear together with ions of intermediate charge states [13].

It is well known that when a multiply charged ion gets close to a solid surface, electron capture processes occur. The captured electrons populate highly excited Rydberg states on the projectile, and subsequently decay to lower lying states by Auger process or radiative de-excitation leading to UV or X-ray emission [14]. The identification of the non-deflected particles as photons is affirmed by the fact that such emission is observable only for tilted samples, i.e. when a significant fraction of the incoming ions are impinging on the capillary walls.

4. Conclusions

We have measured the angular and charge state distribution of Ne^{6+} ions transmitted through alumina capillary samples by different methods. By using a dispersive electrostatic spectrometer, angular distribution of ions with well known charge states has been determined, while the full two-dimensional distribution of all the emitted particles has been exposed by the position sensitive MCP imaging technique. Clear guiding effect has been found for both studied samples at 3 and 6 keV ion impact energies. Moreover, the parameters for the guiding effect determined by the different methods have agreed within the experimental error. A more detailed experimental work is in progress. In the incoming beam, a strong photonic component was observed, but it could be separated from ions by an electrostatic deflector. An important new experience is that for tilted capillaries, photons originated from the capillaries were also observed. Their contribution can be significant, especially for large tilt angles. This means that special care must be taken to separate them from the ions in ion guiding experiments by MCP detectors.

Acknowledgments

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